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<b>(21) International Application Number:</b> PCT/EP99/05045 <b>(22) International Filing Date:</b> 15 July 1999 (15.07.99)  <b>(30) Priority Data:</b> 60/093,903                      23 July 1998 (23.07.98)                      US  <b>(71) Applicant:</b> SOCIETE DES PRODUITS NESTLE S.A. [CH/CH]; P.O. Box 353, CH-1800 Vevey (CH).  <b>(72) Inventors:</b> BORLAND, Carol; 30425 Alder Road, Richwood, OH 43344 (US). CHENG, Pu-Sheng; 6952 Literary Lane, Dublin, OH 43017 (US). LANTIN, Nora; 4099 Leirim Court, Dublin, OH 43016 (US).  <b>(74) Agent:</b> McCONNELL, Bruce; 55, avenue Nestlé, CH-1800 Vevey (CH).		<b>(81) Designated States:</b> AU, BR, CA, CN, IN, JP, KR, MX, NO, NZ, RU, SG, ZA, European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE).  <b>Published</b> <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
<b>(54) Title:</b> LIQUID COFFEE PRODUCT  <b>(57) Abstract</b>  A ready-to-serve coffee product which provides a refreshing beverage or topping. The product is a black, coffee containing liquid in an aseptically filled container. The liquid has a fresh, clean taste and may contain more than 2 % by weight of soluble coffee solids. The liquid is produced by extracting coffee solids from roast and ground coffee at a draw-off ratio of less than about 3. The concentration of soluble coffee solids in the coffee extract is then adjusted to less than about 5 % by weight under oxygen-reduced conditions.		

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### Liquid Coffee Product

5 This invention relates to a liquid coffee product which may provide a refreshing beverage or topping. The invention also provides a method for the production of the liquid coffee product.

Ready-to-serve, coffee-based beverages are becoming increasingly popular. Very often, these beverages contain milk solids or a whitener. Apart from providing a beverage having an appearance and taste similar to that of whitened coffee, the milk solids or whitener performs the function of masking some of the  
10 harsher flavours which may arise in ready-to-drink coffee beverages. However, when served chilled, coffee beverages which contain milk solids or a whitener are perceived to be less refreshing than black coffee beverages.

Ready-to-serve black coffee beverages are available; especially in Asian markets. However, to avoid harsh flavours, these beverages contain less than 2%  
15 by weight of soluble coffee solids. Also, these are usually heavily sweetened. Despite this, these beverages are often still characterised by harsher, dirty flavours and often do not provide refreshing beverages when consumed chilled.

It is therefore an object of this invention to provide a black, ready-to-serve, coffee-based product which has a fresh, clean flavour.

20 Accordingly, in one aspect, this invention provides a ready-to-serve coffee product comprising;

an aseptically filled container; and

a black, coffee containing liquid in the container; the liquid having a fresh, clean taste.

25 Preferably, the liquid has a coffee solids concentration of above about 2% by weight; more preferably above about 2.2% by weight.

In a further aspect, this invention provides a ready-to-serve coffee product comprising;

an aseptically filled container; and

30 a black, coffee containing liquid in the container; the liquid having a fresh, clean taste and a concentration of soluble coffee solids above about 2.2% by weight.

In another aspect, this invention provides a ready-to-serve coffee product comprising a black, coffee containing liquid in a container; the liquid having a  
35 fresh, clean taste, and wherein the liquid is produced by a process comprising:

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extracting coffee solids from roast and ground coffee at a draw-off ratio of less than about 3 to produce a coffee extract;

adjusting the concentration of soluble coffee solids in the coffee extract to less than about 5% by weight under oxygen-reduced conditions for providing the coffee containing liquid.

Preferably, the coffee containing liquid is aseptically filled into the container.

In yet another aspect, this invention provides a method for providing a chilled coffee beverage, the method comprising providing an aseptically filled container containing a black, coffee containing liquid; the liquid having a fresh, clean taste and a concentration of soluble coffee solids above about 2.2% by weight;

opening the container; and

pouring the black, coffee containing liquid over ice particles.

In a further aspect, this invention provides a dessert topping product which comprises;

an aseptically filled container; and

a black, coffee containing liquid in the container; the liquid having a fresh, clean taste and a concentration of soluble coffee solids above about 2.2% by weight.

The coffee containing liquid may contain a sweetener and may contain flavours.

Embodiments of the invention are now described by way of example only. This invention provides a liquid, black coffee product which has a fresh, clean taste with little objectionable bitter and harsh notes and which is in ready-to-serve form. The liquid coffee product may contain high amounts of soluble coffee solids; for example above 2% by weight. In general, conventional black, liquid coffee products which are provided in ready-to-serve form have a soluble coffee solids content of about 1% and below. Concentrations of soluble coffee solids above about 1.5% by weight generally cannot be used in conventional products since the product would then have objectionable harsh and bitter notes.

The liquid, black coffee product is produced by preparing a coffee extract. The coffee extract is prepared by subjecting roasted coffee beans to extraction under mild temperature conditions and using a low draw-off ratio. The roasted coffee beans are preferably ground prior to extraction. Any suitable beans or

blends or beans may be used. Naturally, the better the bean quality, the better the product finally produced.

The extraction may be carried out in a counter-current manner in one or more extraction vessels. Any suitable extraction vessels may be used; for  
5 example fixed bed reactors or continuous counter-current extractors. The choice and design of the vessels is a matter of preference and has no critical impact on the process. Further, if fixed bed reactors are used, the extraction liquid may be caused to flow upward through the reactor or downward through the reactor, as desired. However, the extraction is conveniently carried out in a battery of fixed  
10 bed reactors connected such that extraction liquid may flow through them in series.

The extraction is carried out under relatively mild conditions such that little or no hydrolysis of the coffee solids takes place. Therefore, the temperature of the extraction liquid used to extract the coffee solids is preferably below about  
15 140°C; and more preferably below about 120°C.

The extraction liquid is conveniently hot water which may be, but need not necessarily be, deoxygenated. Coffee extract may also be used as the extraction liquid but this is less preferred.

The draw-off ratio for the extraction is preferably less than about 3:1; more  
20 preferably less than about 2:1. It is surprisingly found that extraction using low draw-off ratios provides a coffee extract with a cleaner, fresher taste with fewer harsh notes.

The coffee extract obtained will usually have a concentration of coffee solids in the range of about 8% by weight to about 15% by weights. The coffee  
25 extract is maintained under reduced oxygen conditions. If desired, oxygen scavengers or antioxidants such as ascorbic acid may be added to the coffee extract. If it is necessary to store the coffee extract for any significant length of time, storage is best conducted under refrigerated conditions.

The coffee extract is then formulated to provide the liquid coffee product.  
30 Ingredients such as sweeteners, flavours, buffers and the like may be added and the concentration of the soluble coffee solids reduced to the desired level. Ordinarily, the concentration of the soluble coffee solids will be adjusted to about 0.8% to about 3.5% by weight; more preferably about 2% to about 3% by weight.

If used, examples of suitable buffers include sodium and potassium  
35 bicarbonate, di-sodium and di-potassium phosphates, and sodium and potassium

citrates. Various combinations of these buffers may also be used. The buffering system used will depend largely upon regulations in the various countries.

Further, if used, examples of suitable sweeteners include sugar, artificial sweeteners such as saccharin, cyclamates, acetosulfame, sucralose, L-aspartyl  
5 based sweeteners such as aspartame, and mixtures of sugar and artificial sweeteners.

Suitable flavours which may be used include vanilla, bourbon, whisky, chocolate, and the like.

The mixture of all components may then be subjected to homogenisation if  
10 necessary. Oxygen-reduced conditions are preferably used during formulation of the liquid coffee product.

The homogenised mixture is then preferably subjected to ultra high temperature (UHT) treatment. This may be accomplished by rapidly heating the mixture to a temperature above about 135°C; for example to about 140°C to  
15 about 150°C. This may be carried out in a suitable heat exchanger or by direct injection of steam into the liquid beverage mix. Suitable heat exchangers are plate heat exchangers, tubular heat exchangers and scraped surface heat exchangers; plate heat exchangers being particularly suitable.

The mixture is held at the raised temperature for a time less than about 2  
20 minutes but sufficient to provide a  $F_0$  value of at least about 4. Depending upon the temperature to which the mixture is heated, the mixture is conveniently held at the temperature for about 5 seconds to about 60 seconds. The  $F_0$  value reached is preferably about 5 for products which will be cold vended. For products which will be hot vending, the  $F_0$  value may be about 60 to 75.

25 The treated mixture is then cooled to a temperature below about 60°C; for example to less than about 40°C. This may be conveniently accomplished by using a suitable heat exchanger or by flash cooling the beverage. Again a plate heat exchanger is a particularly suitable heat exchanger. Flash cooling is especially suitable if the mixture was heated using direct injection of steam since  
30 the introduced steam may then be flashed off. Otherwise the increase in water content must be accounted for when preparing the mixture.

If needed and not added earlier, suitable buffers and other additives may be may be added at this point.

The liquid coffee product obtained is then aseptically filled into suitable  
35 containers. Standard aseptic filling machinery may be used. The containers are

then sealed and stored. The aseptic filling is preferably carried out under reduced oxygen conditions.

If desired, although less preferable, the liquid coffee product may be filled into suitable containers and then retorted. In this case, aseptic filling is not  
5 necessary. The filling is preferably carried out under reduced oxygen conditions. Standard filling and retorting machinery may be used.

The products obtained are characterised by a fresh, clean taste with little of the harsh, bitter notes of most conventional ready-to-drink black coffee beverages. This is despite the fact that the product may contain raised levels of  
10 soluble coffee solids. The products may be consumed in many different ways. For example, the product may be poured over crushed or shaved ice to provide a refreshing beverage. Alternatively, the product may be used as a topping on desserts such as ice cream. Further, the product may be frozen and served as a dessert; especially when mixed with other frozen desserts or fresh whole milk.

15 It is found that the products contain an advantageous ratio of aldehydes to furans of less than about 1:12; more preferably about 1:4 to about 1:10. It is believe that these characteristics have a favourable impact on the flavour of the products.

In this specification, the term "draw-off ratio" means the ratio of the weight  
20 of the coffee extract removed from the extraction system to the weight of fresh coffee introduced into the extraction system.

The term "F<sub>0</sub> value" means the equivalent time in minutes for a product held at a temperature of 121°C.

In this specification, the term "Furans" means compounds of the furan class  
25 including 2-methylfuran.

The term "Diketones" means aroma compounds of the diketone class including 2,3-butanedione, and 2,3-pentanedione.

The term "Aldehydes" means aroma compounds of the aldehyde class including isobutyraldehyde, 3-methyl-butanal, and 2-methyl-butanal.

30 The term "Pyrroles" means compounds of the pyrrole class including 1-methyl-1H-pyrrole.

The term "ppm methyl butyrate" means the calculated amount of a compound based upon the response factor of methyl butyrate.

Specific examples of the invention are now described to further illustrate  
35 the invention. In the examples, aroma components are analysed using gas chromatography and mass spectrometry. The aroma components are introduced



into the gas chromatograph using a purge and trap method (no static headspace). The components separated in the gas chromatograph are detected using a mass spectrometer. The conditions of the gas chromatograph and mass spectrometer are as follows:

5

Column	Restek RTX-1 60m x 0.25mm x 1.0 $\mu$ m
Flow rate	20 ml/minute in He carrier gas
Split ratio	20:1
Initial temperature	35°C
Initial hold time	1 minute
Temperature increase rate	4°C per minute
Final temperature	190°C
Solvent delay	0 minutes
Scan range	35 to 260 amu
Electron energy	70 volts.

All results are expressed in ppm methyl butyrate units.

#### Example 1

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An extraction battery of three fixed-bed extractors is used. The extractors are connected in series such that an extraction liquid may flow into a first extractor, to the second extractor, to the third extractor, and then out of the third extractor. The third extractor contains a charge of about 27 kg of fresh Arabica coffee grounds. The second extractor, which was the third extractor in a previous cycle, contains partially extracted coffee grounds. The first extractor, which was the second extractor in a previous cycle, contains the most extracted coffee grounds.

15

Water at about 104°C is introduced into the first extractor as the extraction liquid. The draw-off ratio is about 1.7:1 to provide about 45 kg of coffee extract. The extraction yield is about 20% and the concentration of coffee solids is about 11% by weight. The coffee extract is held in a container sized such that little or no head-space is present.

20

The coffee extract is diluted to a coffee solids concentration of about 1% by weight under reduced oxygen conditions. Buffering agents are added and the

25



coffee product is aseptically filled into packs containing about 250 ml. Reduced oxygen conditions are maintained.

The coffee product has a fresh, clean taste with perceived nutty and roasty notes.

5

#### Example 2

The coffee extract of example 1 is diluted to a coffee solids concentration of about 1% by weight under reduced oxygen conditions. Sugar (about 5% by weight) and buffering agents are added and the mixture is aseptically filled into packs containing about 250 ml. Reduced oxygen conditions are maintained.

10

The coffee product has a fresh, clean taste with perceived nutty and roasty notes.

15

#### Example 3

A pack produced according to example 1 is opened and the coffee product is then analyzed for diketones, aldehydes, furans, and pyrroles. The results are as follows:

20

Compound	Product of Example 1 Amount (ppm methyl butyrate)
Diketones	0.2
Aldehydes	1.2
Furans	0.1
Pyrroles	0.08
Isobutyraldehyde	0.4
3-methyl-butanal	0.4
2-methyl-butanal	0.4

#### Example 4

A pack produced according to each of examples 1 and 2 is opened and the coffee product is then analysed for acids. The acid break down of each coffee product is as follows:

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Acid	Product of example 1 Amount mg/g	Product of example 2 Amount mg/g
Quinate	1.37	1.09
Acetate	0.28	0.20
Glycolate	0.12	0.10
Formate	0.14	0.11
Chloride	0.03	0.02
Malate	0.11	0.09
Fumarate	0.01	0.02
Sulfate	0.07	0.03
Oxalic	0.01	0.00
Phosphate	0.07	0.07
Citrate	0.25	0.25

The pH of each product is 5.9. The product of example 1 has a turbidity of 14.00 NTU while the product of example 2 has a turbidity of 32.00 NTU. These results indicate that the product are relatively clear.

5

#### Example 5

10 A coffee extract is produced as described in example 1 and is diluted to a coffee solids concentration of about 2.5% by weight under reduced oxygen conditions. Buffering agents, sugar and honey are added and the coffee product is aseptically filled into packs containing about 250 ml. Reduced oxygen conditions are maintained.

The coffee product has the following constituents:

Substance	Amount (weight %)
Water	75.405
Sugar	18.93
Coffee solids	2.5
Honey	3.0
Sodium Bicarbonate	0.165

The coffee product is then analysed for diketones, aldehydes, furans, and pyrroles. The results are as follows:

5

Compound	Product of Example 5 Amount (ppm methyl butyrate)
Diketones	0.2
Aldehydes	1.2
Furans	0.1
Pyrroles	0.05
Isobutyraldehyde	0.4
3-methyl-butanal	0.4
2-methyl-butanal	0.4

The pH of the coffee product is about 6.2 to 6.4. The coffee product is poured over crushed ice to provide an iced beverage. The ice beverage has a fresh, clean taste with perceived nutty and roasty notes. The beverage is refreshing with no perceivable bitter and harsh notes despite the high concentration of coffee solids.

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#### Example 6

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The coffee product of example 5 is poured over vanilla ice cream and presented to a taste panel as a dessert. The dessert is readily consumed with none of the panel finding objectionable bitter and harsh notes.

Claims

1. A ready-to-serve coffee product comprising;  
an aseptically filled container; and  
5 a black, coffee containing liquid in the container; the liquid having a fresh,  
clean taste.
2. A coffee product comprising a black, coffee containing liquid in a  
container; the liquid having a fresh, clean taste, and wherein the liquid is  
10 produced by a process comprising:  
extracting coffee solids from roast and ground coffee at a draw-off ratio of  
less than about 3 to produce a coffee extract;  
adjusting the concentration of soluble coffee solids in the coffee extract to  
less than about 5% by weight under oxygen-reduced conditions for providing the  
15 coffee containing liquid.
3. A product according to claim 2 in which the coffee containing liquid is  
aseptically filled into the container.
- 20 4. A product according to claim 3 in which the coffee containing liquid is  
thermally treated at a temperature above 135°C to obtain an  $F_0$  value of at least 4  
prior to aseptic filling.
5. A ready-to-serve coffee product comprising;  
25 an aseptically filled container; and  
a black, coffee containing liquid in the container; the liquid having a fresh,  
clean taste and a concentration of soluble coffee solids above about 2.2% by  
weight.
- 30 6. A dessert topping product which comprises;  
an aseptically filled container; and  
a black, coffee containing liquid in the container; the liquid having a fresh,  
clean taste and a concentration of soluble coffee solids above about 2.2% by  
weight.

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7. A product according to any of claims 1 to 5 in which the liquid has a coffee solids concentration of above about 2% by weight.
8. A product according to any of claims 1 to 6 which has a ratio of aldehydes to furans of less than about 1:12.
9. A product according to any of claims 1 to 6 which has a ratio of aldehydes to furans of 1:4 to 1:10.
10. A method for providing a chilled coffee beverage, the method comprising providing an aseptically filled container containing a black, coffee containing liquid; the liquid having a fresh, clean taste and a concentration of soluble coffee solids above about 2.2% by weight; opening the container; and pouring the black, coffee containing liquid over crushed or shaved ice.

# INTERNATIONAL SEARCH REPORT

International Application No  
PCT/EP 99/05045

**A. CLASSIFICATION OF SUBJECT MATTER**  
IPC 7 A23F5/24

According to International Patent Classification (IPC) or to both national classification and IPC

**B. FIELDS SEARCHED**

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 A23F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

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Y	page 227 -page 229	10
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☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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International Application No

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## C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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